Rapid Technique for Solvent Removal in the Determination of Fat

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A rapid technique for solvent removal in the determination of fat is described. The official 60 min procedure is reduced to 4–10 min by the use of a nitrogen gas manifold and an aluminum cooling chamber. The precision of the technique is found to be as acceptable as the official method.

Official direct fat analysis of meat (1) requires the following periods of time: 15 min for cutting and grinding 3 times; 20 min for preparation and weighing; 90 min drying at 125°C; 240 min extraction with solvent; 10 min reclaiming solvent by distillation; 30 min drying at 100°C; 30 min cooling; and 5 min weighing, a total of 440 min or 7 hr and 20 min.

In this laboratory, in determinations using the official method, we found that a 30 min drying time at 100°C was sufficient to remove solvent from a Goldfisch beaker. To remove solvent completely from a 250 ml flask required from 40 to 60 min of drying time at 100°C. The average cooling time of the beakers or flasks was 30 min inside a metal desiccator. This total time of a minimum of 60 min for solvent removal and cooling was reduced to 4–10 min by the method described here. This technique was a prerequisite to developing a rapid ether extraction procedure presently being investigated for analysis of meat products.

Experimental

Beef ground 3 times through a ⅛″ meat cutting plate was analyzed by the official method (1). After a 4 hr ether extraction on the Goldfisch apparatus the solvent was removed from the extracted fat by replacing the sample thimble with a reclaiming tube and evaporating almost to dryness. The amount of solvent remaining in the beaker before drying the fat

was ca 0.5 ml. The beakers containing the extracted fat were dried 30 min at 100°C, cooled for 30 min in a metal desiccator, and weighed.

The fat in the beakers from the above experiment was used in obtaining the data presented in this paper regarding the effectiveness of a rapid technique. Ether was added to the residual extracted fat in amounts of 1 to 3 ml. The beakers were rotated manually until the fat was dissolved in the ether. The amount of ether added simulated the maximum possible range of solvent remaining in the beaker after the reclaiming procedure. The solvent was removed by nitrogen drying, utilizing a 6-place gas manifold with individual control valves mounted on a Goldfisch fat extraction apparatus (Fig. 1). The beakers were positioned on the swing beaker holders. The high heat control was used (435°C), with the hot plate touching the swing beaker holder. The stream of dry, clean nitrogen gas was directed at the fat and solvent with the flow controlled so that the fat was not lost from the beaker. In addition to aiding removal of ether, the nitrogen gas cools the fat below the burning point. A period from 3 to 7 min, depending on the volume of solvent present, was used for nitrogen drying under high heat. The heat and nitrogen controls were turned off. The beakers were cooled in an aluminum cooling chamber (Fig. 2). The chamber was constructed 4″ thick, bored to a 3″ depth and 2.1″ diameter (maximum size of a Goldfisch beaker). The overall diameter of the chamber was 8″; a T-shaped detachable handle facilitated fitting it into a metal desiccator. The duration of cooling the beakers was 3 min in a chamber equilibrated at room temperature or 1 min in a prechilled chamber (0 to −20°C).

A warning should be noted for analysts using ethers that may form peroxides. Drying should be done on a steam bath or boiling water bath. Ovens and other electrical or flame heating devices should be avoided when possible and the drying should be done in a hood with adequate safety shields in use.

Results and Discussion

Table 1 shows the effect of high heat and nitrogen drying on the removal of solvent added to the residual fat extracted by the official method.

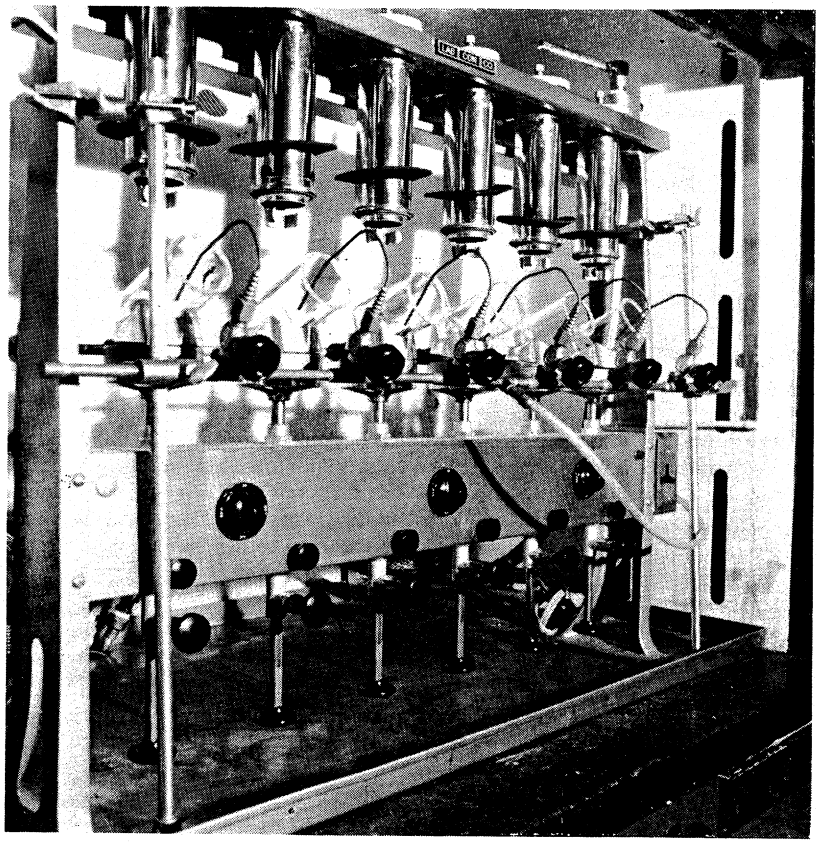

FIG. 1—Nitrogen gas manifold mounted on 6-place Goldfisch extraction apparatus.

Table 1. Effect of high heat and nitrogen drying on removal of solvent of residual fat extracted by the official method

Sample	Ether Added, ml	High Heat-N_2 Drying, min	Original Sample Wt, g	Change in Wt of Residual Fat, 10^{-3} g	% Fat Error[a]
1	3	7	2.4795	−3.0	−0.12
2	3	7	3.8015	−1.9	−0.05
3	3	7	2.6100	−1.3	−0.05
4	3	7	3.1890	−1.6	−0.05
5	3	7	2.6074	−1.3	−0.05
6	3	7	3.9993	−0.4	−0.01
7	2	5	3.0572	+0.9	+0.03
8	2	5	2.9854	+0.3	+0.01
9	2	5	3.9623	−0.4	−0.01
10	2	5	2.9752	−0.3	−0.01
11	1	3	2.0464	−0.2	−0.01
12	1	3	4.8979	+0.5	+0.01
13	1	3	3.4233	−1.7	−0.05
14	1	3	2.4879	−0.5	−0.02
				Mean error	−0.03

[a] % fat error = (change in wt of residual fat, g ÷ original sample wt, g) × 100.

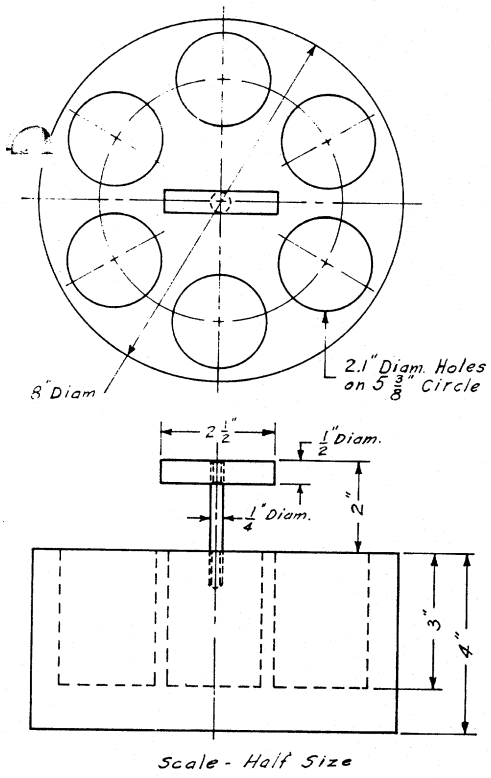

FIG. 2—Aluminum cooling chamber for Goldfisch beakers.

In discussion of the meaningful error of fat analysis there is no published definition of accuracy, other than it is generally recognized that the official method (1) is empirical. Precision is generally acceptable if the difference between duplicates does not exceed 0.5% fat. The 0.5% level has also been proposed by the International Organization for Standardization (2). The results indicate that reduction in time of analysis from 60 min minimum to 4–10 min maximum for removal of solvent and cooling does not change the range of precision expected from this method.

Acknowledgments

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References

(1) *Official Methods of Analysis*, 10th Ed., AOAC, Washington, D.C., 1965, secs. **22.033** and **23.005**.

(2) International Organization for Standardization, Subcommittee ISO/TC 34 SC 6, Working Group 2: "Second Draft ISO Proposal for the Determination of the Fat Content of Meat and Meat Products," April 1964.